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1,3:4,6-Bis(1,4-diphenylethynyl-2,3-xylylene)tetrahydro-3a,6a-bis(ethoxycarbonyl)imidazo[4,5-*d*]imidazole-2,5(1*H*,3H)-dione, a molecular clip based on di(ethoxycarbonyl)glycoluril

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Key indicators

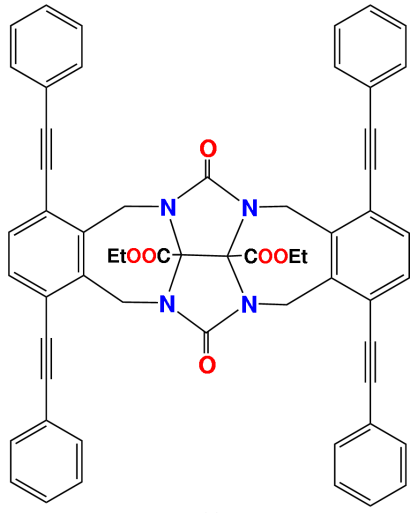
Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.005 Å
R factor = 0.062
wR factor = 0.174
Data-to-parameter ratio = 13.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The molecule of the title compound, $C_{58}H_{42}N_4O_6$, a molecular clip based on the glycoluril framework, occupies a special position on a twofold axis passing through the mid-point of the gycoluril C—C bond. The planes of the central benzene rings in the opposite wings of the molecular clip form a dihedral angle of 41.6 (4)°. No dimers typical for molecular clip packings are observed in the crystal structure of the title compound.

Comment

A molecular clip is a molecule with a rigid U-shaped cavity, in which small aromatic guest molecules can be complexed by hydrogen-bonding and aromatic stacking interactions (Rowan *et al.*, 1999). The glycoluril skeleton has served as an important building block for the preparation of a wide variety of supramolecular assemblies, including molecular clips (Rowan *et al.*, 1999; Wu, Chakraborty *et al.*, 2002), molecular capsules (Hof *et al.*, 2002), cucurbit[*n*]uril derivatives (Freeman *et al.*, 1981; Lagona *et al.*, 2003) and, most recently, anion-binding receptors (Kang *et al.*, 2004). Recently, we introduced a molecular clip (Wu, Fettinger & Isaacs, 2002) based on the title concave di(ethoxycarbonyl)glycoluril, (I). In this paper, we report the results of the X-ray diffraction study of this diethoxycarbonyl glycoluril derivative.



(I)

The molecule of (I) occupies a special position on a twofold axis passing through the mid-point of the glycoluril bond C26—C26^{i} [symmetry code: (i) $\frac{1}{2}-x,\frac{3}{2}-y,z$] (Fig. 1). The C9–C14 and C9^{i}–C14^{i} benzene rings in the opposite wings of the clip form a dihedral angle of 41.6 (4)° with each other. The

plane of the central C9–C14 ring forms dihedral angles of 58.5 (2) and 11.0 (1)° with the peripheral rings C1–C6 and C17–C22, respectively, whereas the planes of the C1–C6 and C17^{i}–C22^{i} rings, coming close to each other spatially but belonging to the different wings of the molecular clip, are almost orthogonal [dihedral angle 92.4 (2)°].

There are no π–π stacking interactions in the crystal structure of (I). No formation of dimeric aggregates, typical for the packing of other molecular clips, is observed in this structure.

Experimental

The title compound was synthesized according to the procedure reported by Wu, Fettinger & Isaacs (2002). Crystals of (I) suitable for X-ray data collection were obtained by slow evaporation of a dichloromethane solution at 283 K.

Crystal data

$C_{58}H_{42}N_4O_6$
M_r = 890.96
Orthorhombic, *Pccn*
a = 22.0779 (17) Å
b = 11.0552 (9) Å
c = 18.9962 (15) Å
V = 4636.5 (6) Å^3
Z = 4
D_x = 1.276 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3666 reflections
θ = 2.3–20.0°
μ = 0.08 mm^{-1}
T = 292 (2) K
Block, colourless
0.20 × 0.20 × 0.10 mm

Data collection

Bruker SMART 4K CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
25639 measured reflections
4109 independent reflections
2611 reflections with $I > 2\sigma(I)$
R_{int} = 0.067
θ_{max} = 25.0°
$h = -24 \rightarrow 26$
$k = -13 \rightarrow 13$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.174
S = 1.05
4109 reflections
309 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0821P)^2 + 1.2253P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

All H atoms were initially located in a difference Fourier map. Methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.93 Å and U_{iso}(H) = 1.5U_{eq}(C), but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.96–0.97 Å and with U_{iso}(H) = 1.2U_{eq}(C).

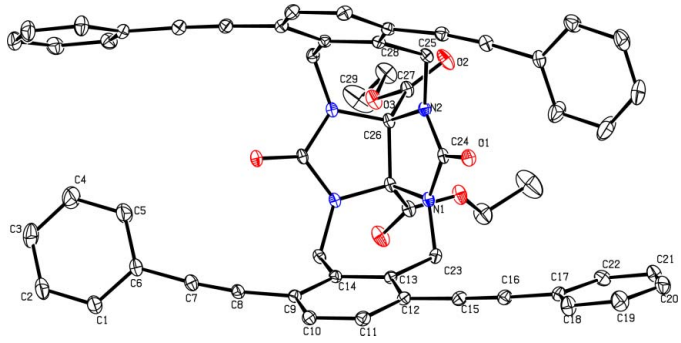


Figure 1
A view of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted. Unlabelled atoms are related to labelled atoms by ($\frac{1}{2} - x$, $\frac{3}{2} - y$, z).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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